



Triboelectrostatic separation of fly ash and charge reversal[☆]

John P. Baltrus*, J. Rodney Diehl, Yee Soong, William Sands¹

U.S. Department of Energy, National Energy Technology Laboratory, P.O. Box 10940, Pittsburgh, PA 15236-0940, USA Received 19 April 2001; revised 25 September 2001; accepted 25 September 2001; available online 5 December 2001

Abstract

Triboelectrostatic separation has been investigated as a method for separating unburned carbon from coal combustion fly ash. It was found that when a fly ash is exposed to moisture before it undergoes separation, the charging properties of the components of the fly ash change significantly. The mineral and carbon components of the fly ash appear to charge oppositely to how they were charged before exposure to moisture. A correlation was found between the degree of charge reversal and the relative amounts of leachable ions, especially calcium and sodium ions, present on the surface of the ash. © 2001 Elsevier Science Ltd. All rights reserved.

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1. Introduction

The presence of significant amounts of unburned carbon in coal-combustion fly ash can be attributed to inefficient combustion in the boilers of older power plants, and more recently is a consequence of the use of low- NO_x burners to reduce power plant emissions. The carbon must be removed from these fly ashes so that they meet the requirements for maximum loss-on-ignition (LOI) imposed by the users of fly ash in industries such as cement manufacturing [1]. Otherwise the fly ash must be disposed in a landfill at a significant cost to the utility. Additionally, if isolated in sufficient purity, the carbon portion of the fly ash may also be a valuable by-product when used directly as filler or as a sorbent following activation [2–4]. Dry triboelectrostatic separation has been studied and commercially applied for the removal of unburned carbon from coal-fired utility fly ash [5,6].

Some form of electrostatic separation has been employed in the separation of ores and minerals for almost a century [7]. A specific type of electrostatic separation called triboelectrostatic separation has been used to purify a variety of materials including minerals, coal and more recently, fly ash [5,8–10]. The basic process relies on the triboelectrification of the materials that need to be separated through contact and exchange of electrical charges between the materials themselves or with another dissimilar solid. While much

is known about triboelectrification, there is still a significant lack of knowledge about the basic mechanisms affecting the charging of insulators [7,11].

Triboelectrostatic separation of fly ash is usually accomplished by triboelectrification of the fly ash followed by passing it between two oppositely charged electrodes. A typical fly ash that has not been exposed to moisture will charge so that the carbon-enriched component will be attracted to or deposited on the negative electrode and the ash-enriched component will be attracted to or deposited on the positive electrode. An exception to this behavior has been noted when dealing with fly ash that at some time has been exposed to moisture [12,13]. In the latter case, the carbon and ash-rich fractions report to electrodes opposite to which they would be attracted had they not been exposed to moisture. Fly ash can be exposed to moisture during handling if it is temporarily stored in a sluice pond or experiences high humidity. While most fly ash is typically handled so that it is not exposed to moisture, exposure to moisture is common enough that its consequences are important to those who wish to separate such fly ash using triboelectrostatic separation. Exposure to moisture could cause unwanted behavior of the carbon and ash fractions leading to a decrease in the overall efficiency of fly ash separation.

The mechanism by which fly ash undergoes triboelectrification is not understood enough to easily allow explanation of the factors that control charge reversal following exposure to moisture. In one study of charge reversal, it was suggested that charge reversal is predominantly affecting the carbon and not the ash [12,13]. There has been some investigation of how triboelectrostatic separation of fly ash

^{*} Corresponding author. Tel.: 412-386-4570; fax: 412-386-4806.

E-mail address: john.baltrus@netl.doe.gov (J.P. Baltrus).

¹ Current address: Ametek, Inc., Process and Analytical Instruments Division, 150 Freeport Road, Pittsburgh, PA 15238-3493, USA.

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Table 1
Triboelectrostatic separation data for fly ashes and synthetic mixtures of fly ashes with graphite

Sample	$\% \ C_{feed}$	- Cyclone			+ Cyclone		
		Weight (g)	% C	% of total C	Weight (g)	% C	% of total C
Fly ash A							
As-received	16.0	32.3	37.2	71	67.7	7.2	29
Exposed to moisture	15.8	67.0	9.0	40	27.0	33.4	60
Treated pH 9.3	15.7	65.3	6.5	29	34.7	30.1	71
Treated pH 3.6	15.8	45.1	20.3	56	54.9	12.7	44
850 °C + graphite	11.7	26.3	37.6	94	59.2	1.1	6
850 °C + graphite exposed to moisture	11.6	22.0	29.7	80	45.7	3.5	20
Fly ash B							
+ graphite	11.6	41.2	20.9	81	51.8	4.0	19
+ graphite exposed to moisture	11.7	51.7	1.8	11	24.2	32.2	89
850 °C + graphite exposed to moisture	12.4	70.7	2.2	14	22.6	43.0	86

is affected by the chemical and physical properties of the ash [5,14]. The present study focuses on the charge reversal phenomenon and what properties of the ash can be altered in order to minimize charge reversal.

2. Experimental

2.1. Samples

Fly ash was obtained from two utilities burning bituminous coals. One fly ash (A) contained approximately 16% carbon and came from a boiler fitted with low-NO_x burners. The other fly ash (B) came from a boiler fitted with conventional burners and contained virtually (less than 1%) no carbon.

2.2. Sample treatments

In order to investigate whether the pH of the moisture has an effect on triboelectrostatic separation, 100 g of fly ash A were mixed with just enough of a pH 9.3 sodium tetraborate buffer to form a thick slurry. The slurry was then dried at 110 °C. The same procedure was used to treat another 100 g sample of fly ash A with a pH 3.6 KHP-HCl buffer. Finally, a few grams of fly ash were mixed with non-buffered deionized water in a glass petri dish to create a thick slurry. The sample was then dried at 110 °C to remove the water.

In another set of experiments, synthetic mixtures of fly ash containing a known type and amount of carbon were prepared in order to better understand the role of carbon in triboelectrostatic separation and charge reversal. Samples of fly ash A and fly ash B were placed in a muffle furnace at room temperature. The temperature was then raised to 850 °C and held for 5 h. This temperature was found in another study to be sufficient to remove all of the carbon from the fly ash [15]. Although fly ash B initially contained virtually no carbon, the heating treatment was carried out for comparison purposes. The heat-treated fly ashes were then mixed with graphite powder (Alfa, -325 mesh) to yield 100 g of mixture containing approximately 12%

carbon. The mixtures were separated by triboelectrostatic separation, recombined, exposed to moisture under the conditions described above, and then reseparated.

2.3. Triboelectrostatic separations

A parallel plate separator was used for this study [14] and all separations were conducted in air. The separator consisted of a venturi feed system driven by pressurized nitrogen gas, an injection nozzle, and a high voltage separation section. The fly ash particles pass through the venturi feeder and become charged in this turbulent flow by contact with the copper tubing and with one another. The contact of the particles with copper surfaces, especially in the turbulent zone of the in-line static mixer, results in effective charging of both unburned carbon and the mineral matter. The charged particles are then forced out of the nozzle in a ribbon of entrained particles between two parallel charged plates 15.24 cm long and 7.62 cm apart. The electric field voltage is +25,000 volts. A splitter is placed downstream between the ends of the charged plates to separate the carbon-rich and ash-rich fractions and direct them to two collection cyclones. The separator has a capacity of 8 kg/h in continuous operation and typically 95% or more of the fly ash that is fed into the system is recovered in the two collection cyclones. For this particular separator very little ash attaches to the electrodes.

2.4. Sample analyses

The carbon contents of the starting materials and separated fractions were measured in duplicate by using a carbon analyzer. X-ray photoelectron spectroscopy (XPS) was used to look for differences in the surface compositions of the as-received and treated ashes. Elemental surface concentrations were calculated from peak areas based on manufacturer-provided sensitivity factors. The XPS instrument employed monochromatic Al $K\alpha$ X-rays and the pass energy of the analyzer was 58.7 eV.

The quantity of soluble ions present in some fly ash

Table 2 Surface elemental concentrations (% atomic) for fly ash A before and after exposure to moisture and buffer solutions of different pH. ND = not detected

Element	As-received	Exposed to moisture	Treated pH 9.3	Treated pH 3.6	
С	35.2	40.0	40.3	37.5	
O	47.6	43.9	38.5	44.5	
Al	3.6	3.8	3.3	4.1	
Si	9.1	8.8	6.7	9.3	
S	2.0	ND	ND	ND	
P	0.51	0.82	0.49	1.5	
Na	0.48	0.18	2.1	ND	
Ca	0.62	1.0	1.1	0.34	
K	ND	ND	ND	0.50	
В	ND	ND	5.2	ND	
Mg	ND	ND	1.7	1.5	
Fe	0.78	0.89	0.66	0.77	

samples was measured by placing 0.1 g of fly ash in 25 ml of deionized water and stirring for 10 min. The solution was then filtered, and the conductivity of the filtrate was measured using a conductivity meter.

3. Results

The results of treatment at different pH on the separation of carbon from fly ash A are reported in Table 1. The amount of fly ash separated was 100 g in each case. The as-received fly ash separated similarly to fly ashes in other studies [12,13], with the carbon-rich fraction being attracted to the negatively charged plate. Charge reversal was evident on exposure to moisture. The relative degree of charge reversal varied with pH. The percentage of carbon in the two cyclones that collected the separated fractions was nearly opposite of what was found before the fly ash was wetted and dried. Treatment with the basic buffer resulted in an even greater fraction of the carbon reporting to the positive electrode compared to the sample wetted with deionized water. Conversely, the fly ash that was treated with an acidic buffer did not undergo charge reversal to the extent experienced by the sample treated with deionized water. Movement of additional carbon to the positive cyclone following exposure to moisture was accompanied by the movement of additional ash from the positive to the negative cyclone.

The elemental surface concentrations of the as-received and treated samples, as determined by XPS, are reported in Table 2. A simple exposure to moisture caused a change in

Table 3
Conductivity data for solutions used to leach soluble ions from fly ash samples

Sample	Conductivity/g ash $(\mu S/g)$		
Fly ash A	515		
Fly ash A - 850 °C	218		
Fly ash B	3110		
Fly ash B - 850 °C	1690		

the surface concentrations of some elements such as sulfur, calcium and sodium. Surprisingly sulfur was not detected on the surface of any of the treated ashes despite the fact that the samples were merely exposed to water or the buffers and then dried without being washed. The most notable changes in surface concentration of elements occurred for the ashes treated with buffer solutions. The ash treated with the pH 9.3 buffer had an appreciable amount of boron and sodium on the surface, probably from evaporation of the buffer solution. The ash treated with pH 3.6 buffer had a somewhat elevated level of potassium, also from the buffer. Both buffer-treated ashes had increased levels of magnesium on the surface.

Data for the synthetic mixtures of fly ash and graphite are presented in Table 1. More of the carbon component of fly ash A that was heated to 850 °C and then mixed with graphite was attracted to the negative fraction than the original carbon that was present in the unheated fly ash. The carbon in the mixture of unheated fly ash B with graphite also separated as would be expected for a typical highcarbon fly ash. It also underwent charge reversal when exposed to moisture before separation. A significant difference in the behavior of the two fly ashes was seen after both were heated to 850 °C, mixed with graphite, exposed to moisture and then separated. Pretreating fly ash B to 850 °C before preparing the mixture with graphite had no significant effect on the degree of charge reversal. However, the degree of charge reversal was greatly diminished for the fly ash A synthetic mixture exposed to moisture compared to the behavior of the as-received fly ash.

The quantities of soluble ions in fly ashes A and B were measured before and after heating to 850 °C. The measurement was indirect, as what was measured was the effect of all the soluble ions on the conductivity of a solution containing the fly ash. The solution conductivities are reported in Table 3 and were normalized to the quantity of ash (e.g. carbon was excluded) in each fly ash sample. Comparatively, fly ash A had significantly fewer soluble ions than fly ash B. The amount of soluble ions decreases about 50% after the heat treatment.

The elemental surface concentrations of the two fly ashes

Table 4 Surface elemental concentrations (% atomic) for fly ashes A and B before and after heating at 850 $^{\circ}\text{C}$ for 5 h

Element	Fly Ash A	Fly Ash A 850 °C	Fly Ash B	Fly Ash B 850 °C
С	35.2	19.7	28.3	27.7
O	47.6	55.5	49.8	48.9
Al	3.6	6.7	2.8	3.8
Si	9.1	15.2	10.3	10.3
Na	0.48	0.40	3.9	1.4
Ca	0.62	0.63	3.8	5.7
Fe	0.78	1.3	1.2	2.3

before and after heating to 850 °C were determined by XPS and are reported in Table 4. Only the major elements common to all the samples were measured. Even though oxidation at high temperature removed all of the bulk carbon, as determined by bulk analysis, and fly ash B contained only trace amounts of bulk carbon, significant surface concentrations of carbon were detected. The surface carbon was adventitious carbon resulting from normal exposure of the material to air. Oxidation at 850 °C resulted in little change in the surface concentration of Na and Ca on fly ash A. There was a significant decrease in carbon concentration due to oxidation and removal of unburned carbon from fly ash A. For fly ash B, the only changes observed upon treatment at 850 °C were the surface concentrations of Na and Ca.

4. Discussion

The data for the as-received fly ash A are consistent with previous reports of charge reversal during triboelectrostatic separation of fly ash that has been exposed to moisture, indicating that charge reversal is not an isolated phenomenon. In order to help understand charge reversal, fly ash containing no carbon can be combined with graphite to yield samples that behave similarly to fly ash containing unburned carbon.

The unburned carbon in fly ash typically resembles amorphous graphite [16]. Graphite is conductive while the aluminosilicates that make up the bulk of the mineral matter in fly ash are known for their insulating properties. During contact electrification between conducting and insulating particles, negative charge moves from the conductor to the insulator and the conductor, in this case graphite, becomes positively charged [7,11]. The same phenomenon occurs when the insulating ash particles come in contact with the copper metal tubing in the feed line of the triboelectrostatic separator. The greatest separation efficiency can be achieved when the differences in Fermi levels of the participant materials are maximized. Separation of carbon and mineral matter occurs in a triboelectrostatic separator because carbon in fly ash acquires a positive charge and is attracted to the negative plate while the mineral matter acquires a negative charge and is attracted to the positive plate.

The former description is a rather simplistic way of looking at a complicated electrical mechanism for charge exchange. Charge exchange depends not only on relative conductivities of the materials being separated, but more specifically on the surface work functions of the materials, which are affected by particle size, shape, the presence of sharp edges, surface roughness, lattice defects, contamination, temperature, humidity, etc. [7,10,17]. The exchange is not perfect, and that is one of the reasons why complete separation of the components of fly ash does not occur. Perfect separation of carbon and ash into 100% pure components also does not occur because the carbon and ash particles are interlocked and are influenced by the electrical properties of the material making up a majority of the surface of the particle. We have previously examined high-carbon fly ash under a microscope and found particles that were predominantly ash, but contained embedded carbon particles. Also to a lesser degree, particles that were predominantly carbon were observed to contain ash inclusions [16]. Multiple passes of the separated fractions of fly ash can be done in order to increase the purity of the separated products, but this is usually achieved at the expense of overall yield of the pure fractions. For the triboelectrostatic separator configuration used in the present study, the fly ash is required to report to one of the two cyclones used for collection. Other separators use multiple louvers or have a central zone to collect particles that are not charged.

There are a number of areas that require additional research in order to understand the detailed mechanism of charge behavior during triboelectrostatic separation of fly ash. The mechanism described above provides basic reasoning for why a dry fly ash behaves as it does during triboelectrostatic separation. The changes that occur to the fly ash during exposure to moisture and result in a seeming reversal of the charging properties of the components of the fly ash are less clearly understood. Keep in mind that although the ash and carbon concentration numbers for the two collection cyclones seemingly reverse after the fly ash is exposed to moisture, complete charge reversal of the originally separated materials probably does not occur after they are exposed to moisture. Rather, when working with a fixed amount of fly ash, a portion of the carbon that was predisposed to charging positively is now charging negatively, while a corresponding portion of the ash has also reversed charge and is now charging positively.

On a weight basis, the exchange between cyclones is not equal. Up to approximately 40 g of ash moves from the positive cyclone to the negative cyclone after exposure to moisture while from 2 to 5 g of carbon reverses charge. One reason for this is that charge exchange between carbon and ash is dependent on contact area. While glassy ash particles can have skeletal densities of up to 50% greater than the unburned carbon particles depending on the petrographic form of carbon [17], they may have bulk densities that are even more than those of the unburned carbon. Therefore

when individual equally sized particles of carbon and ash collide, on a weight basis, a greater exchange of ash will occur. Another reason may be a difference in size of the ash and carbon particles. If a smaller carbon particle collides with a larger ash particle, unequal masses will undergo charge exchange. There are probably other possible scenarios leading to charge exchange that would account for an uneven balance of mass, but as noted earlier the charge transfer process is poorly understood.

At first glance, the data in Table 1 suggest that the pH of the moisture may have some effect on the degree of charge reversal. The effect of pH on triboelectrostatic separation of minerals has been studied previously [18]. In order for charge reversal to occur, a change would be required in the relative electron energy levels of the carbon and ash particles. It was stated above that electrification occurs when charge (electrons) is transferred from conductor to insulator. Under normal conditions, charge moves from the carbon to the ash particles or from the copper tubing in the inlet to the ash. Therefore during charge reversal, the donor of charge must now be the ash. Note that the degree of charge reversal is greater at higher pH. A change in pH would be expected to affect the degree of surface protonation/hydroxylation of the mineral matter in the ash. Such a change may affect the electrical properties of the mineral matter, but it seems that other factors should be involved in order to produce a significant enough change required to make the ash a donor of charge [18].

An examination of the XPS data in Table 2 shows that the pH 9.3 sample, which underwent the greatest degree of charge reversal, also had a significant amount of sodium and boron on the surface. The ions were deposited on the surface when the buffer that was used to treat the fly was evaporated. The pH 3.6 sample, which experienced the least degree of charge reversal, had elevated concentrations of potassium and phosphorus on the surface compared to the other samples, but otherwise had the lowest sodium and calcium surface concentrations. The elevated levels of potassium and phosphorus on the pH 3.6 sample again resulted from evaporation of the buffer used to treat the sample.

The relative amounts and types of ions present on the ash surface can influence the electrification properties of the ash. Previous studies suggest that the work functions of mineral surfaces could be altered by the adsorption of different ions [10,18]. Such preconditioning is used to increase the separation efficiency of the minerals. The ions can be a source of excess charge as well as alter the local conductivity of the ash. Further analyses of the separated fractions by XPS showed that the ions from the evaporated buffer solutions deposited without preference on the ash and carbon particles. However their effects can depend on their local concentrations and their degree of dispersion. Based on a review showing that the surface work function can be reduced by the adsorption of negative ions [10], it is possible that borate ion adsorption on the surface of insulating ash particles may decrease the tendency of those surfaces to

charge negatively. Such was the case when borate ions were deposited on quartz [18]. The ions would be expected to have somewhat less of an effect on the carbon particles because their naturally conductive properties may help mitigate significant changes in surface conductivity.

The possible influences of surface ions on the electrification of ash particles was further demonstrated through the use of synthetic mixtures of graphite powder and ash containing no or trace unburned carbon. The ash with no carbon was obtained by oxidatively removing all of the unburned carbon from fly ash A. A comparison of the separation data for the as-received fly ash A with the data for a synthetic mixture of fly ash A residue with graphite in Table 1 shows that better separation of carbon from the ash was achieved for the latter sample. This is probably because none of the carbon in the synthetic mixture is interlocked with the ash particles, as is typically found with unburned carbon in fly ash.

Although both synthetic mixtures of fly ash and graphite behaved similarly when undergoing triboelectrostatic separation after they are first prepared, their behaviors diverged once they were exposed to moisture. The fly ash B mixture exhibited normal charge reversal once it was exposed to moisture, but very little of the fly ash A mixture underwent charge reversal. It appeared that the oxidation treatment at 850 °C, which was used to remove carbon from the ash, induced a chemical or physical change in the ash that altered its electrical charging properties. Because fly ash B had not been heated prior to mixing with graphite and subsequently exhibited charge reversal, it was thought that charge reversal could possibly be prevented by pretreating that fly ash at 850 °C prior to mixing it with graphite. However the results reported in Table 1 show that the heat pretreatment had little effect on the charge reversal properties of the fly ash B mixture. In order to eliminate differences in bulk chemical composition or particle sizes as the reason for a difference in behavior of the heat-treated fly ashes with respect to charge reversal, the ashes were examined with X-ray diffraction, infrared spectroscopy and scanning electron microscopy. No significant differences were found in bulk composition, or particle morphology and size distributions of the heat-treated ashes.

The role of surface ions with respect to charge reversal was reexamined in light of the results obtained using the synthetic mixtures. This was done indirectly by measuring the 'availability' of the ions to become mobile once the ash was placed in an environment of excess moisture. The mobile ions described here would be analogous to the surface ions described earlier in the pH experiments. It was hypothesized that the conductivity of an aqueous solution containing the ash could be related to the ability of the soluble ions to affect surface charge during triboelectrification.

The conductivities for solutions containing fly ashes A and B, which are reported in Table 3, are significantly different both before and after treatment at 850 °C. There was a significant percentage decrease in conductivity of the

solutions after the ashes were treated at 850 °C. Compared to fly ash A, a solution containing fly ash B had a significant conductivity value even after the ash was treated at 850 °C. The availability of a significant number of 'mobile' ions in fly ash may be the reason it easily undergoes charge reversal even after the heat treatment. The case of fly ash A is not as clear. If mobile ions play a role in charge reversal, then there must be a threshold in the concentration of mobile ions needed for charge reversal somewhere in the range required to yield solution conductivities between 515 and 218 $\mu S/g$ ash. The number of mobile ions needed to reach the onset of charge reversal and whether other factors may also play a role requires further study.

The surface concentrations of ions on fly ashes A and B before and after treatment at 850 °C were measured using XPS. There does not appear to be a significant enough change in the surface concentrations of Na and Ca following heat treatment, as reported in Table 4, to explain the measured change in conductivities of solutions containing the fly ashes. It is possible that the heat treatment could have changed the structure of the surface of the ash without affecting the relative concentrations of certain elements like Na and Ca. Specifically, heating at 850 °C over 5 h may have brought about the beginning of a phase transition to a structure where the Na and Ca are bound more tightly to the aluminosilicate matrix. This could affect the electrical properties of the ash by decreasing the mobility of the ions or simply inducing a change in surface energy levels through changes in bonding. There is a significant difference in the relative surface concentrations of Na and Ca when comparing fly ash A with fly ash B in Table 4. The concept that surface ions play an important role in charge reversal is supported by the observation that fly ash B, which still exhibited charge reversal after heating at 850 °C, had the higher surface concentrations of Na and Ca. A significant number of surface ions in fly ash B were still soluble after the heat treatment as evidenced by the conductivity results in Table 3.

5. Conclusions

The results of this study have practical applications for the triboelectrostatic separation of unburned carbon from fly ash. Foremost, the results indicate that because charge reversal is governed by the mobility of surface ions when the fly ash is exposed to moisture, it is difficult to inhibit. The most logical preventive measure is not to expose the fly ash to moisture. This is not always practical and would probably increase handling costs. As demonstrated above, another method that can prevent charge reversal in some ashes is heat treatment in order to lock the mobile ions within the surface structure of the ash. This also is not practical because the high temperatures required to lock the mobile ions in the ash structure would lead to oxidative removal of the remaining carbon and therefore no separation would be required. The results above suggest that the best method for separating carbon from fly

ash that has been exposed to moisture is to treat those ashes further by the addition of salts containing sodium, calcium and borate ions. The salts would maximize charge reversal and fly ashes treated with those salts could be processed separately from dry fly ashes. The separated fractions would be equivalent to those for a dry fly ash, but they would be collected on opposite electrodes. The net result would be the same as for the separation of a fly ash that was never exposed to moisture.

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